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Section B-2

Investigation of the Melting Point of RDX (OD-12)

by Frank C. Whitmore

OSRD No. 538

Serial No. 233

April 30, 1942

Endorsement (1) from F. C. Whitmore, Chairman, Section B-2 to Roger Adams, Chairman, Division B.

> "The results and conclusions presented in this report should be considered when new melting point specifications are set for Bachmann RDX."

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NATIONAL DEFENSE RESEARCH COMMITTEE

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OFFICE OF SCIENTIFIC RESEARCH AND DEVELOPMENT

Report on "Investigation of the Melting Point of RDX"
to
April 20, 1942
by
F. C. Whitmore
Pennsylvania State College

OSRD No. 538

Serial No. 233

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Date: April 30, 1942

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"The results and conclusions presented in this report should be considered when new melting point specifications are set for Bachmann RDX."

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INVESTIGATION OF THE MELTING POINT OF RDX

The Pennsylvania State College

April 20, 1942

ABSTRACT

A description of apparatus and technique for a suggested standardized melting point determination especially designed for the Bachmann product is presented. The most important suggestion is that the lower value of the melting point range be the temperature at which frothing first occurs.

Samples of two batches of RDX were sent to nine laboratories with the request that each determine the melting points by their usual procedure. Each received portions of the same sample A and sample B. The results of these determinations are included.

DISCUSSION AND EXPERIMENTAL

The only practical method for determing the purity of a sample of RDX, with the possible exception of a carbon analysis, is by an accurate melting point determination. In the case of a compound such as RDX this determination is unusually difficult for a number of reasons. The material decomposes on melting. This prevents a true equilibrium between the liquid and solid phases, thus making the melting point dependent on the rate of heating. For this reason the change should probably be called a decomposition point (dp)rather than a melting point. Also, any melting point of the order of 200°C., if taken in the conventional melting point apparatus may be subject to a correction up to 6° depending on the thermometer used.

Since the product from the Bachmann process is probably a mixture of RDX, m.p. 204°, and HMX, m.p. 278°, the melting point determination is unusually subject to these difficulties.

The British specification for RDX reads as follows:

"Melting point (with decomposition): not below 200° C. (corr.).

tube
Determined in a melting point to be attached to a thermometer in an air bath surrounded by a stirred liquid, the temperature of which, from 190°C. is raised at the rate of 0.5° to 1.0°C. per minute."

The method of correction is not indicated. We are sure that when a correction is used its magnitude and method of calculation should be given. The British specification was designed for direct nitration product for which it is satisfactory since the melting point occurs rather sharply at 204-204.5° for pure RDX. In the case of the Bachmann product, however, the following changes occur on heating (see below for details of temperature readings). At about 185-190°, the solid in the capillary shrinks slightly. At about 193-5° it takes on a waxy appearance and appears to soften. At about 195-7° the material froths and portions are forced up the capillary tube. During this stage bubbles are liberated in the molten portions. At about 193-201° the RDX has completely melted.

The apparatus used in determining the melting points included in this report consisted of an air bath 1.1 cm. in diameter and 45 cm. in length. The air bath was surrounded by a stirred bath of chlorinated diphenyl (Monsanto Aroclor 1248), (4.0 cm. in diameter and 35 cm. in length) which was heated by a nichrome wire (size 25, 2.07 ohms/ ft., 30 ft., 42 turns) wound full length around the outside and controlled by a Variac (type 200 C). The thermometer to which the capillary was attached was a total immersion type, 0-250°, with 0.5° divisions, which had been calibrated for total immersion by the Bureau of Standards. No correction was necessary since the thermometer was totally immersed in the heated air bath. A drawing of the apparatus is included.

The capillaries which were used were about 0.04" diameter and were fastened to the thermometer and placed in the air bath when the liquid was at such a temperature that the thermometer reading rapidly rose to 170-180°.

Melting points of various samples of direct nitration product are listed below.

#1 - Direct nitration product fumed-off and recrystallized from acetone 204-204.5°

#2 - Direct nitration product fumed-off 204-204.5°

#3 - Direct nitration product fumed-off, dissolved in acetone, treated with Norite, filtered, small amount of concentrated ammonium hydroxide added, cooled. 204-204.5°

#4 - Direct nitration product fumed-off, recrystallized from 1-nitropropane. 204-204.5°

#5 - Direct nitration product fumed-off, recrystallized from glacial acetic acid. 204-204.5°

#6 - Direct nitration product prepared at Picatinny Arsenal by direct nitration, collected by diluting, filtering, drying, recrystallizing from acetone. Made in 1926. Crystals stored dry until November 1940. Shipped under water. Filtered and air dried. 203.5-204.5°

#7 - Direct nitration product fumed-off, heated for three hours at 80° with a 5% sodium carbonate solution. 204-204.5°

#8 - Direct nitration product fumed-off, heated with water at 80° for 15 hours. 204-204.5°

#9 - Same as sample #8. Time of heating 30 hours. 204-204.5°

#10 - Same as sample #8. Time of heating 60 hours. 204-204.5°

#11 - Authentic sample of British RDX received from du Pont 204-204.5°

#12 - Authentic sample of British RDX obtained from Bruceton Testing Laboratories. 203.8-204.0°

Melting points of various samples of product by methods other than direct nitration are listed below. The softening point (in brackets) is the temperature

at which the material had acquired a definite waxy appearance. The lower value in each melting point range is the point at which frothing first occurs. This figure is important since it is known that RDX does not decompose appreciably in the solid state at elevated temperatures for short periods. Therefore, frothing indicates some melting accompanied by the usual decomposition. The taking of this temperature as the start of the melting point range had the added advantage that the first point of frothing is a definite point in an otherwise indefinite transition. The point of complete melting is difficult to determine and may be largely a matter of opinion.

#13 -	Product from procedure Johnson CU-3, run at 75-78°, filtered at 60°, first crop	(193)	196-198°
#14 -	Sample #13 recrystallized from 60% nitric acid	(198)	200.5-202°
#15 -	Sample #13 recrystallized from acetone.	(200)	201-202°
#16 -	Second crop from same procedure as #13. After heating in diluted filtrate.	(180)	185.5-195°
#17 -	Sample #16 recrystallized from 60% nitric acid	(183)	186-190°
#18 -	Product from procedure Johnson CU-3 except that reaction was run at 55-58°. First crop collected at 55°.	(191)	195.5-197°
#19 -	Sample #18 recrystallized from 60% nitric acid	(198)	199.5-201°
#20 -	Sample #18 recrystallized from acetone	(198)	199-200.5°
#21 -	Second crop from procedure described in #13. After heating in diluted filtrate	(186)	189-193°
#22 -	Product from procedure Johnson CU-3 collected in one crop run at 75-78°.	(186)	190-197°
#23 -	Sample #22 recrystallized from 60% nitric acid	(190)	193-196°
#24 -	Sample #22 recrystallized from acetone.	(198)	199-201.5°

#25 - Product from procedure Johnson CU-3 collected in one crop run at 55-58° (194) 197-198°

#26 - Sample #25 recrystallized from 60% nitric acid. (195) 197-200.5°

#27 - Sample #25 recrystallized from acetone. (200) 201.5-202.5°

Results on samples sent to different laboratories.

Samples of two batches of RDX were sent to each of the laboratories listed below with the request that each determine the melting points by their usual procedure. Each received portions of the same sample A and the same sample B.

Sample A Direct nitration product, fumed-off, and heated for 30 hours with distilled water at 85°.

Sample B Product from procedure Johnson CU-3 recrystallized from 60% nitric acid. Johnson CU-3 is a modified Bachmann procedure in which the hexamine is added dissolved in glacial acetic acid.

Cornell University -

Sample A 204-204.5° corr. The correction applied in these
Sample B 197-197.5 Corr. cases amounted to 5°.

"These melting points were taken in our usual procedure using a thermometer that had been checked against an Anschutz verified at 206° by boiling nitrobenzene. The samples were immersed at about 170° and the rate of heating was 3 to 3.5° per minute."

University of Michigan -

Sample A as received (204) 204.5-205°

Sample B as received (190) 196-201°

Finely ground (201) 201.5-204°

Screened:
On 200 mesh (ground) (201) 202-204°
Through 200 mesh (190) not completely melted at 230°.

Western Cartridge Company:

Sample A	Froth 204.4°	Melted 205°
Sample B	Froth 199.5°	Melted 202°

"These melting points were run:

1. In a vacuum jacketed air bath.
2. Heated by forced circulation of oil.
3. At a carefully controlled rate of 1° a minute.
4. Using a Taylor short-stem thermometer #1077066 graduated in .1°C.
5. At total immersion.
6. The sample being inserted in the air bath with the bath at a temperature of 190°C.

We have found that the melting point is extremely sensitive to moisture, and hence all samples should be vacuum dried. However, these tests were run on material as received."

Tennessee Eastman Corporation -

Sample A as received		203.8-204.6° No previous softening.
Sample B	ground	(199.5) 200.4-202°

"Sample A Particles are non-uniform both as to size and shape but consist mostly of rhomboids and rhomboidal fragments. Melting point as received 203.8 to 204.0°C. with no previous softening.

After being ground in a mortar the observed melting point was 204 to 204.5°C.

Sample B consisted mainly of plates, although a number of fines of irregular rhomboidal shapes were observed. These plates were too large to enter our melting point tube as received so the sample was ground in a mortar and the melting point was found to be 200.4° to 202°C. with previous softening at 199.5°C.

A sample of the fine material was screened from Sample B and the material passing 100 mesh softened at 190°C. and melted at a range of 191.6 to 199°C.

The Pennsylvania State College -

Sample A 204-204.5°

Sample B (193) 197.5-200°

A small portion of sample B was ground in an agate mortar and melting points taken on the material with capillaries of 0.03", 0.04", 0.05" and 0.06" diameter. In all cases the material melted at 197.5-201°. A sample of the original material in a capillary 0.05" in diameter also melted at the same point. The capillaries ordinarily used in this laboratory have a diameter of about 0.04". The capillaries Dr. Homer Adkins sent to various laboratories as a suggested standard also were about 0.04" in diameter.

University of Toronto:

Sample A Melted 204.5-205° with almost no prior softening when inserted in the rising bath at 50°, 130°, or 160°.

Sample B Melted at 193.5° and 199.5° (two portions of the same sample, melting points taken simultaneously) when inserted at 50°, at 201.5° when inserted at 130° and 203° when inserted at 160°.

du Pont Company -

Sample A (201.6°) 202.8-203.4°C.

Sample B (194.5°) 196.2-201.1°C.

The temperature of the bath was raised about 0.5°C./min.

Harvard University -

Sample A (201.5°) 202.5-203° Frothing at 202.5°

Sample B (190°) 196-202° Frothing from about 200°

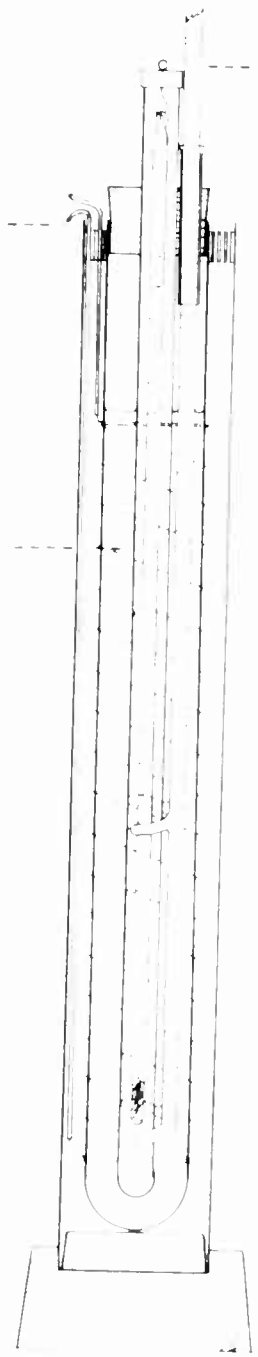
These were taken both in an air bath and in an acid bath. The thermometers were calibrated Anschutz and the values given are corrected for stem exposure. The rate of heating was approximately that given in the specification. The values above were found in the air bath. The values found in an acid bath (Hershberg apparatus) run about a degree higher than the above in all respects.

McGill University -

Sample A 203.5-204°

Sample B (191)197-200°

In each case five operators took the melting point and the above values are those selected as being most typical.



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